MONITORING THE PERSISTENCE AND MOVEMENT OF FENAMIPHOS IN SOILS OF LILY BULB FIELDS IN DEL NORTE COUNTY, 1986

MARCH, 1988

CDFA

ENVIRONMENTAL HAZARDS ASSESSMENT PROGRAM

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by

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ABSTRACT

Three lily bulb fields, one in each of three areas of the Smith River Plains in Del Norte County, were monitored in 1986 for the persistence and downward movement of fenamiphos that had been applied to the fields in the fall of 1985. In two of the areas where aldicarb had contaminated the ground water, fenamiphos residues were found at increasingly greater depths in soil cores collected 5 months and again 9-10 months after application. In a third area where bulbs had never been previously grown, similar results were found. Deep soil-coring in that field in August showed fenamiphos residues present 9 feet deep in the soil profile. Fenamiphos was present at shallower depths in samples collected in December, about 14 months after application. The parent compound as well as the sulfoxide and sulfone breakdown products were present in some portion of each soil core that was collected. Soil and environmental conditions present in the areas studied appear to favor persistence of fenamiphos residues and permit downward leaching of the pesticide over time.

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Disclaimer

The mention of commercial products, their source or use in connection with material reported herein is not to be construed as either an actual or implied endorsement of such product.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

In 1983, aldicarb (Temik®) and 1,2-D (1,2-dichloropropane, a component of D-D soil fumigant) were found as contaminants in the Smith River Plains ground water basin located in Del Norte County (1). The two chemicals were used extensively as nematicides in the lily bulb industry located there. The contamination was discovered during a study initiated in 1983 by the North Coast Regional Water Quality Control Board (NCRWQCB); well sampling is still being continued in the study area. The Smith River Plains area was selected for study because it was considered a high risk area due to the combination of coarse soils, a shallow ground water table, high rainfall and heavy use of soil-applied pesticides.

Although the use of aldicarb and 1,2-D have been suspended in Del Norte County, the same conditions exist for potential contamination of the ground water by other pesticides. Preplant soil fumigation of lily bulb fields is now accomplished with Telone II®, a product containing 1,3-dichloropropene and minute quantities of 1,2-D. Fenamiphos (Nemacur®) has replaced aldicarb as a nematicide applied to the root zone at the time bulbs are planted in raised beds. During the past two years, well water samples collected from the study area by the NCRWQCB have been analyzed for fenamiphos with no positive finds to date (2).

Because of the potential threat to ground water by the increased application of fenamiphos, the Environmental Hazards Assessment Program (EHAP) of the California Department of Food and Agriculture (CDFA) began cooperative work with the NCRWQCB in March, 1986. The purpose of this work was to study the persistence and movement of fenamiphos in the soils of Del Norte County lily bulb fields. This report summarizes the results of soil monitoring conducted from March through December, 1986.

MATERIALS AND METHODS

Sampling Area

The NCRWQCB defined four study areas in the Smith River Plains for the purpose of well sampling (Figure 1, courtesy of Sue Warner, NCRWQCB). Areas A, B and C were selected because of known ground water contamination problems; area D, a new bulb farm operation located near the Smith River, represented a potential threat to ground water.

We visited bulb growers in February, 1986, and obtained permission to collect soil samples from one field each in study areas A, B and D. The fields had been treated with fenamiphos in the fall of 1985 at the time bulbs or bulb scales were planted. In preparation for planting, the soil was prepared as raised beds spaced 42 inches apart. The bulbs were planted in a furrow about one foot wide in the center of the bed and fenamiphos (Nemacur 10G) granules were then applied in a 10-12 inch band over the bulbs at rates varying from 60 to 120 lbs per acre based on 12,445 feet of row per acre. Fenamiphos granules were typically located at a depth of 4 to 8 inches below the soil surface after application.

Soil Sampling

Soil samples were collected from each of the three fields in March, July and August, 1986, approximately 5, 9 and 10 months, respectively, after fenamiphos had been applied. The fields were harvested during September and October, 1986.

The soil samples collected in March were taken with a one inch diameter Veihmeyer tube or with a 2 1/2-inch diameter bucket auger. The gravelly nature of the soil in the fields we sampled made the use of a Veihmeyer tube difficult; only five samples were collected with it in March. All subsequent sampling was done with a bucket auger which provided larger soil samples that could also be used for physical characterization. The Veihmeyer tube was washed thoroughly with

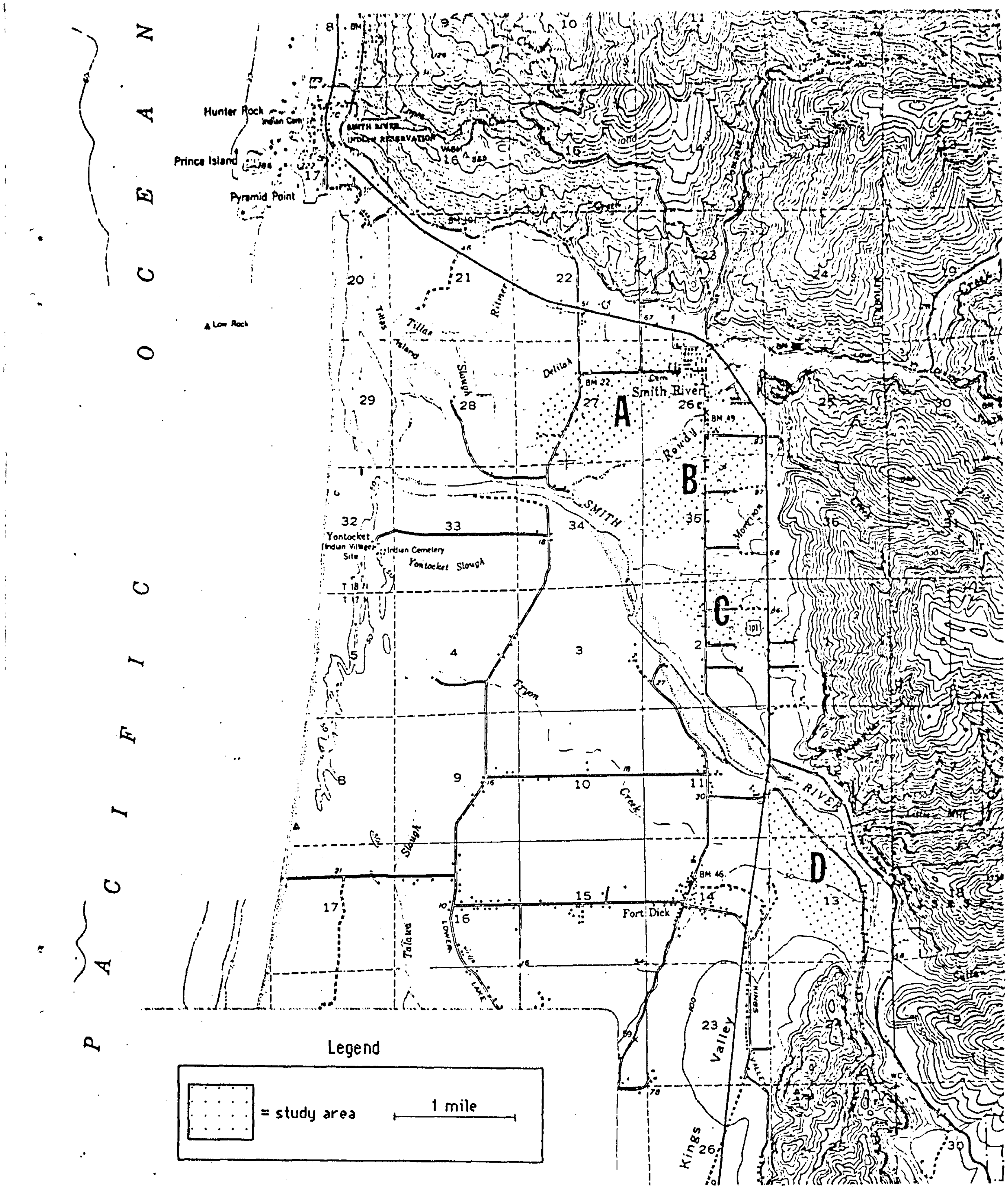

Figure 1. Location of study areas in the Smith River Plains of Del Norte County.

laboratory grade detergent and water, rinsed with tap water and rinsed with isopropyl alcohol between each sample collected. The bucket auger was thoroughly washed in soapy water and rinsed twice with tap water after each sample was collected and was rinsed with isopropyl alcohol before a new soil core was begun.

One or two soil cores were collected from each field on each sample date. However, as the study progressed, additional cores were taken from location D in August and in December. The sites selected for sampling were located approximately 50 feet in from the edge of the field on the end nearest the access road. Areas of the field that were the least gravelly were also selected to prevent plugging or damage to the sampling apparatus. A spot in the center of a planting bed where no plants were located was chosen and the first sample was collected from the surface to 6 inches deep and then at 6 inch intervals. Extreme care was taken not to knock soil from the surface layers back into the hole as sampling progressed. The depth of the hole was measured after each soil sample was removed. Information including the location, date, and depth for each sample was recorded on a chain of custody form. The form accompanied the sample throughout storage and analysis.

A rubber mallet was used to knock excess soil from each end of the bucket auger after it was pulled up from the ground. This was done to remove soil that might have fallen into the open hole during sampling. The remaining soil was knocked loose into a clean polyethylene bag, shaken and homogenized in the bag, and poured into a one pint mason jar until full. The jar was sealed with aluminum foil and a screw cap. Samples were frozen on dry ice immediately after collection and were kept frozen during transport and storage.

At the time the samples were analyzed for fenamiphos, a determination of moisture content was made and results were reported on a soil dry weight basis.

Additionally, measurements of soil pH, organic matter content, and texture were made for soil cores collected from each field in August, 1986.

Laboratory Methods

All chemical analyses for fenamiphos were performed by the CDFA Chemistry Laboratory Services Branch. The analytical method (Appendix I) consisted of a 50:50 (V/V) hexane:acetone extraction with the extracts evaporated to dryness and redissolved in ethyl acetate. The ethyl acetate extracts were then analyzed using a Varian Vista 6000 gas chromatograph (GC) with a thermionic specific detector and a 10 m x 0.53 mm 50% phenyl methyl silicone column. The GC was run at 120-240°C. The remainder of each extract was evaporated to dryness and redissolved in 20:80 (V/V) acetonitrile:water. These samples were analyzed with a Perkin Elmer Series 4 high pressure liquid chromatograph (HPLC) with a 220 nm Spectroflow UV detector and 25 cm x 4.6 mm Sepralyte CH column.

The GC and HPLC analyses were used in conjunction to identify and quantify fenamiphos and its metabolites. Analysis by GC yielded a distinct peak for fenamiphos and a second peak for the combined sulfoxide and sulfone concentrations, therefore, GC analysis was used to quantify fenamiphos concentrations. HPLC produced three peaks corresponding to fenamiphos, fenamiphos sulfoxide, and fenamiphos sulfone. The HPLC peak served as a confirmation for fenamiphos. For the metabolites (sulfoxide and sulfone), the HPLC peaks were used for quantification and GC peaks were used for confirmation. The minimum detectable level for each of the three compounds was 0.01 ppm throughout the study. Quality control procedures, conducted throughout the course of laboratory analyses, consisted of one blank and one spike run with every set of samples.

Measurements of soil pH, organic matter content and particle size analysis were performed by EHAP staff. For soil pH, a 10 gram subsample of soil was mixed with 10 ml of distilled water in a 50 ml beaker, stirred thoroughly with a glass rod and then allowed to stand for at least 30 minutes. The soil suspension was stirred again just before the pH reading was recorded. Organic matter content of soil samples was determined using dichromate reduction (Appendix II). The hydrometer method was used for particle size analysis of soil samples (Appendix III).

RESULTS

Area A

The soil in this field was a dark gray, well drained soil classified as a Rowdy gravelly clay loam with 0-3% slope. One core was taken to a depth of 42 inches in this field in March. Fenamiphos was detected in samples collected from the surface to a maximum depth of 30 inches (Table 1). The maximum concentration detected of 1.45 ppm was found in the 6-12 inch depth. Most samples contained the parent compound and both oxidation products.

Two cores were collected to a depth of 60 inches in July. The first core contained fenamiphos in the top 18 inches of soil, with all three compounds present (Table 1). In the second core, all three compounds were also detected in the upper 18 inches and the fenamiphos parent compound was found 18-24 inches deep and again at 48-54 inches.

Results for the two cores collected in August showed that total concentrations of fenamiphos increased dramatically and that fenamiphos and oxidation products were leaching deeper into the soil beneath the zone of application (Table 1). All three compounds were detected in every sample down to the 42 inch depth in the first core and down to the 48 inch depth in the second core. The highest

Table 1. Concentrations of fenamiphos residues in soil core samples collected three different times after application from a lily bulb field located in Area A.

Approximate depth (inches)	Fenamiphos (ppm) expressed as total residue and fenamiphos (F), sulfoxide (SO), and sulfone (SO2)											
	March 3 (5 month post)[a]				July 15 (9 month post)							
	Core 1				Core 1				Core 2			
	Total	F	SO	SO2	Total	F	SO	SO2	Total	F	SO	SO2
0 - 6	0.49	0.27	0.22	ND[b]	0.42	0.30	0.04	0.08	1.56	0.49	0.84	0.23
6 - 12	1.45	0.09	0.91	0.45	0.23	0.06	0.04	0.13	0.98	0.09	0.44	0.45
12 - 18	0.35	0.02	0.20	0.13	0.13	0.02	0.04	0.07	0.57	0.05	0.34	0.18
18 - 24	0.48	0.05	0.27	0.16	ND	ND	ND	ND	0.02	0.02	ND	ND
24 - 30	0.04	ND	0.02	0.02	ND	ND	ND	ND	ND	ND	ND	ND
30 - 36	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
36 - 42	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
42 - 48	--[c]				ND	ND	ND	ND	ND	ND	ND	ND
48 - 54	--				ND	ND	ND	ND	0.02	0.02	ND	ND
54 - 60	--				ND	ND	ND	ND	ND	ND	ND	ND
% of Total		15	58	27		49	15	36		22	51	27

a. Approximate time post application.
b. None detected; minimum detectable level was 0.01 ppm.
c. Not sampled.

Table 1. (Continued)

Approximate depth (inches)	Fenamiphos (ppm)							
	August 19 (10 month post)							
	Core 1				Core 2			
	Total	F	SO	SO2	Total	F	SO	SO2
0 - 6	1.14	0.30	0.73	0.11	0.37	0.07	0.20	0.10
6 - 12	4.97	3.79	0.61	0.57	3.28	2.50	0.36	0.42
12 - 18	1.73	1.19	0.34	0.20	2.12	1.60	0.32	0.20
18 - 24	0.52	0.20	0.21	0.11	0.65	0.51	0.10	0.04
24 - 30	0.11	0.05	0.05	0.01	0.17	0.09	0.06	0.02
30 - 36	0.09	0.04	0.04	0.01	0.12	0.04	0.05	0.03
36 - 42	0.09	0.04	0.04	0.01	0.17	0.03	0.11	0.03
42 - 48	--				0.07	0.01	0.04	0.02
48 - 54	--				--			
54 - 60	--				--			
% of Total		65	23	12		70	18	12

concentrations were present in the 6-12 inch samples and concentrations generally decreased with increasing depth.

The percentages of parent compound present in total fenamiphos residues generally increased from March to July and August; percentages of fenamiphos sulfoxide showed a general decrease during the same period.

Area B

The soil in this field was classified as a Russ silt loam with 0-3% slope; Russ soils are moderately well to imperfectly drained silt loams. One core was taken from this field in March using a Veihmeyer tube, and two cores were taken in July and August using a bucket auger. In March, samples were collected at 6 inch intervals from 0-12 inches and then at 12 inch intervals from 12-48 inches. This was done to reduce the number of samples to be analyzed. All three fenamiphos compounds were found in the top 12 inches; only the two oxidation products were present in the 12-24 inch segment (Table 2). No soil was collected in the tube at the 24-36 inch depth due to plugging, and fenamiphos was not detected in the next segment, 36-48 inches deep. The highest concentration was found in the 6-12 inch sample.

In the first core collected on July 1, fenamiphos residues were detected in the upper 24 inches of soil and again at 42-48 inches, the deepest sample (Table 2). The second core was taken to a depth of 60 inches and fenamiphos residues were detected only in samples from the upper 30 inches. The fenamiphos parent compound was found in the top 18 inches; the sulfoxide and sulfone were present in all positive samples.

For the two cores collected in August, fenamiphos was found in all segments of the first core to the maximum sample depth of 42 inches, and in all segments of

Table 2. Concentrations of fenamiphos residues in soil core samples collected three different times after application from a lily bulb field located in Area B.

Approximate depth (inches)	Fenamiphos (ppm) expressed as total residue and fenamiphos (F), sulfoxide (SO), and sulfone (SO2)											
	March 3 (5 month post)[a]				July 15 (9 month post)							
	Core 1				Core 1				Core 2			
	Total	F	SO	SO2	Total	F	SO	SO2	Total	F	SO	SO2
0 - 6	1.10	0.30	0.63	0.17	0.89	0.02	0.40	0.47	0.14	0.07	0.03	0.04
6 - 12	2.37	0.09	1.54	0.74	1.30	ND	0.64	0.66	0.50	0.04	0.14	0.32
12 - 18	0.63[b]	ND[c]	0.52	0.11	0.05	ND	0.02	0.03	0.60	0.02	0.19	0.39
18 - 24					0.02	ND	0.01	0.01	0.24	ND	0.13	0.11
24 - 30	LS[d]				ND	ND	ND	ND	0.03	ND	0.02	0.01
30 - 36					ND	ND	ND	ND	ND	ND	ND	ND
36 - 42	ND[e]				ND	ND	ND	ND	ND	ND	ND	ND
42 - 48					0.08	0.08	ND	ND	ND	ND	ND	ND
48 - 54	--[f]				--				ND	ND	ND	ND
54 - 60	--				--				ND	ND	ND	ND
% of Total		9	66	25		4	46	50		9	34	57

a. Approximate time post application.
b. Sample represents a 12 inch long segment, 12-24 inch depth.
c. None detected; minimum detectable level was 0.01 ppm.
d. Lost sample.
e. Sample represents 12 inch segment, 36-48 inch depth.
f. Not sampled.

Table 2. (Continued)

Approximate depth (inches)	Fenamiphos (ppm) — August 19 (10 month post) — Core 1				Core 2			
	Total	F	SO	SO2	Total	F	SO	SO2
0 - 6	0.19	0.15	0.04	ND	3.91	2.64	0.77	0.50
6 - 12	2.42	2.25	0.05	0.12	2.06	1.58	0.18	0.30
12 - 18	1.21	0.92	0.12	0.17	0.32	0.15	0.12	0.05
18 - 24	0.58	0.30	0.16	0.12	0.05	0.05	ND	ND
24 - 30	0.22	0.14	0.05	0.03	0.08	0.05	0.02	0.01
30 - 36	0.04	0.03	0.01	ND	0.01	0.01	ND	ND
36 - 42	0.01	0.01	ND	ND	--			
42 - 48	--				--			
48 - 54	--				--			
54 - 60	--				--			
% of Total		81	9	10		70	17	13

the second core except the deepest one taken at 42 inches (Table 2). The parent compound was present in all positive samples from both cores and was the only compound detected in the deepest sample from both cores. The greatest concentration occurred in the 6-12 inch segment for core one and in the 0-6 inch segment for core two. These concentrations were similar to the concentrations found at this depth in March, more than 5 months earlier.

The percentages of fenamiphos sulfoxide present in total fenamiphos residues decreased from March to July and August; the percentage of parent compound was very high (70 and 81%) in the August samples.

Area D

The soil in this field was classified as a Ferndale fine sandy loam with 0-3% slope. Two cores were collected from an area of the field in March and again in July. In March, fenamiphos parent and the two oxidation products were detected in all samples taken from the top 18 inches of the first core; relatively high concentrations (11.94 ppm) were found in the upper 12 inches of one core (Table 3). Residues were detected in the top 24 inches of the second core and the sulfone was also found at the 30-36 inch depth. No fenamiphos was found at the 36-54 inch samples for either core.

In July, fenamiphos residues were detected at a maximum depth of 42 inches in core one and 48 inches in core two (Table 3). All samples from core two contained the parent compound; the highest concentration was present in the 6-12 inch sample. Both cores contained high total fenamiphos residues (3.99 and 8.83 ppm) in the upper 10 inches of soil.

As mentioned earlier, this field was in a new bulb production area and was under study by the NCRWQCB because of its proximity to the Smith River. Our results

Table 3. Concentrations of fenamiphos residues in soil core samples collected three different times after application from a lily bulb field located in Area D.

	Fenamiphos (ppm) expressed as total residue and fenamiphos (F), sulfoxide (SO), and sulfone (SO2)															
	March 3 (5 month post)[a]								July 15 (9 month post)							
Approximate	Core 1				Core 2				Core 1				Core 2			
depth (inches)	Total	F	SO	SO2	Total	F	SO	SO2	Total	F	SO	SO2	Total	F	SO	SO2
0 - 6	2.80	1.66	1.00	0.14	0.12	0.10	0.02	ND[b]	0.47	0.47	ND	ND	2.30	2.30	IS[c]	IS
6 - 12	9.14	5.14	3.50	0.50	0.62	0.21	0.27	0.14	2.49	1.95	0.27	0.27	5.71	3.87	1.70	0.14
12 - 18	0.62	0.03	0.53	0.06	2.45	1.55	0.78	0.12	1.03	0.18	0.59	0.26	0.82	0.17	0.55	0.10
18 - 24	ND	ND	ND	ND	0.40	ND	0.37	0.03	ND	ND	ND	ND	0.24	0.24	IS	IS
24 - 30	ND	ND	ND	ND	ND	ND	ND	ND	0.02	0.02	ND	ND	0.05	0.05	ND	ND
30 - 36	ND	ND	ND	ND	0.02	ND	ND	0.02	ND	ND	ND	ND	0.02	0.01	0.01	ND
36 - 42	ND	ND	ND	ND	ND	ND	ND	ND	0.04	ND	ND	0.04	0.02	0.01	0.01	ND
42 - 48	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.09	0.09	ND	ND
48 - 54	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	--[d]			
54 - 60	--				--				ND	ND	ND	ND	--			
% of Total		54	40	6		51	40	9		65	21	14		73	24	3

a. Approximate time post application.

b. None detected; minimum detectable level was 0.01 ppm.

c. Not enough soil was available for analysis of SO and SO2 concentrations.

d. Not sampled.

from July suggested that fenamiphos residues had moved deeper into the soil over time and might pose a threat to ground water. Therefore, we conducted a more extensive sampling of this field in August. Five coring sites were selected along three sides of the field (Figure 2). Each site corresponded to an area where the NCRWQCB had taken soil cores to study the soil profile (site maps courtesy of Sue Warner). The cores collected in March and July had been taken from a part of the field corresponding to site #1 on the map. One core was collected from each of the five sites on August 19, 1986. Soil samples were collected to a maximum depth of 108 inches (9 feet) at sites #1, 2, 4 and 5. The core at site #3 was only taken to a depth of 30 inches because the presence of large cobbles and gravel made deeper sampling impossible.

Fenamiphos residues were detected at the maximum sample depth of 102-108 inches in cores from sites #2 and #4, and 84-90 inches at site #5 (Table 4). The deepest positive sample from site #1 occurred at the 48-54 inch depth and at the maximum sample depth of 24-30 inches at site #3. For sites #2 and #4 where the deepest movement of fenamiphos occurred, the highest concentrations were found in the upper 24 inches but residues were found in most of the samples from the surface down. The parent compound was present in every positive sample for these cores and was the only fenamiphos compound present in the 5-8 foot depth. These two sites were located at opposite ends of the field, approximately 750 feet apart.

At site #5, fenamiphos was detected from the surface down to 42 inches and not again until the 66-90 inch zone was reached. The parent compound and the sulfoxide were found in the deepest positive samples. Soil taken at site #1, which was previously sampled in March and July, showed the presence of fenamiphos in most samples from the surface down to 54 inches. The parent compound and sulfoxide were present in all positive samples. At site #3 where samples could only be taken to a depth of 30 inches, fenamiphos residues were detected in all

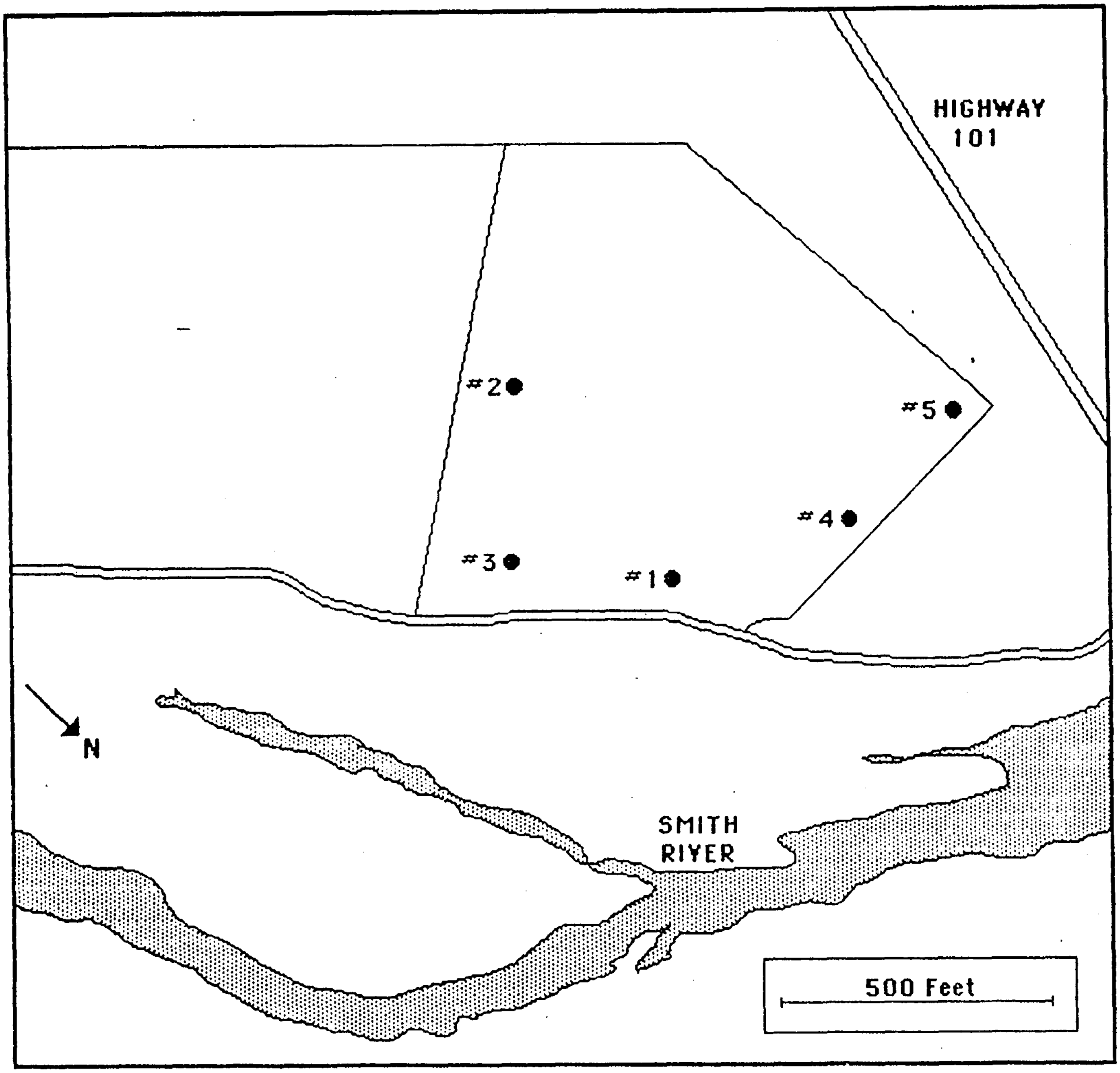

Figure 2. Locations of five sites sampled in August, 1986, in lily bulb field in study area D.

Table 4. Concentrations of fenamiphos residues in soil core samples collected from five different areas of a lily bulb field located in Area D on August 19, 1986, 10 months post application.

Approximate depth (inches)	Core 1 Total	Core 1 F	Core 1 SO	Core 1 SO2	Core 2 Total	Core 2 F	Core 2 SO	Core 2 SO2	Core 3 Total	Core 3 F	Core 3 SO	Core 3 SO2
	Fenamiphos (ppm) expressed as total residue and fenamiphos (F), sulfoxide (SO), and sulfone (SO2)											
0 - 6	0.09	0.04	0.04	0.01	0.13	0.11	0.02	ND[a]	0.04	0.04	ND	ND
6 - 12	0.64	0.26	0.22	0.16	1.17	0.83	0.20	0.14	2.90	1.41	1.00	0.49
12 - 18	0.38	0.18	0.16	0.04	4.62	2.00	2.20	0.42	2.51	1.38	0.60	0.53
18 - 24	2.41	1.05	1.28	0.08	4.85	2.87	1.82	0.16	1.58	0.82	0.60	0.16
24 - 30	0.36	0.16	0.19	0.01	0.38	0.25	0.12	0.01	4.12	2.03	1.76	0.33
30 - 36	ND	ND	ND	ND	0.12	0.05	0.06	0.01	--[b]			
36 - 42	0.03	0.02	0.01	ND	0.15	0.09	0.06	ND	--			
42 - 48	ND	ND	ND	ND	0.05	0.04	0.01	ND	--			
48 - 54	0.06	0.03	0.03	ND	0.05	0.04	0.01	ND	--			
54 - 60	ND	ND	ND	ND	0.01	0.01	ND	ND	--			
60 - 66	ND	ND	ND	ND	0.02	0.02	ND	ND	--			
66 - 72	ND	ND	ND	ND	0.08	0.07	0.01	ND	--			
72 - 78	ND	ND	ND	ND	ND	ND	ND	ND	--			
78 - 84	ND	ND	ND	ND	0.04	0.04	ND	ND	--			
84 - 90	ND	ND	ND	ND	0.01	0.01	ND	ND	--			
90 - 96	ND	ND	ND	ND	ND	ND	ND	ND	--			
96 - 102	ND	ND	ND	ND	0.03	0.03	ND	ND	--			
102 - 108	ND	ND	ND	ND	0.01	0.01	ND	ND	--			
% of Total		44	49	7		56	38	6		51	36	13

a. None detected; minimum detectable level was 0.01 ppm.

b. Not sampled.

Table 4. (Continued)

Approximate depth (inches)	Fenamiphos (ppm)							
	Core 4				Core 5			
	Total	F	SO	SO2	Total	F	SO	SO2
0 - 6	0.02	0.02	ND	ND	0.31	0.16	0.10	0.05
6 - 12	4.90	2.67	1.15	1.08	1.04	0.89	0.10	0.05
12 - 18	2.30	1.20	0.59	0.51	1.41	0.86	0.40	0.15
18 - 24	0.06	0.04	0.01	0.01	0.07	0.02	0.05	ND
24 - 30	0.11	0.05	0.03	0.03	0.02	ND	0.02	ND
30 - 36	0.06	0.04	0.01	0.01	0.04	0.02	0.02	ND
36 - 42	0.04	0.02	0.01	0.01	0.01	0.01	ND	ND
42 - 48	0.04	0.02	0.01	0.01	ND	ND	ND	ND
48 - 54	0.01	0.01	ND	ND	ND	ND	ND	ND
54 - 60	0.05	0.03	0.01	0.01	ND	ND	ND	ND
60 - 66	0.01	0.01	ND	ND	ND	ND	ND	ND
66 - 72	0.01	0.01	ND	ND	0.01	0.01	ND	ND
72 - 78	ND	ND	ND	ND	ND	ND	ND	ND
78 - 84	0.02	0.02	ND	ND	0.07	ND	0.07	ND
84 - 90	ND	ND	ND	ND	0.06	ND	0.06	ND
90 - 96	0.01	0.01	ND	ND	ND	ND	ND	ND
96 - 102	0.01	0.01	ND	ND	ND	ND	ND	ND
102 - 108	0.01	0.01	ND	ND	ND	ND	ND	ND
% of Total		54	24	22		65	27	8

samples; the highest concentrations were present in the 24-30 inch zone. For the three sampling dates, fenamiphos parent made up from 44-73% of the total fenamiphos residue in the cores; fenamiphos sulfoxide 21-49%, and fenamiphos sulfone 3-22% of the total.

Lily bulb fields are normally harvested in September and October, and then plowed in preparation for the next crop. However, due to market conditions for bulbs in 1986, the grower chose not to harvest an area of the field about 2 acres in size. This presented us with an opportunity to conduct additional sampling to determine if fenamiphos was still present in that area of the field and to what depths it may have moved. We collected two cores to a depth of 156 inches (13 feet) on December 4, 1986. The cores were taken from an area located near site #4 (Figure 2).

Fenamiphos residues were present at 0-36 inches deep for one core and 0-30 inches for the second core, indicating that the pesticide persisted in the soil for more than 14 months after application (Table 5). The parent compound and sulfoxide were both present in every positive sample except one. Fenamiphos parent made up 56 and 45%, and fenamiphos sulfoxide made up 25 and 42% of the total fenamiphos residues in the cores. No fenamiphos residues were detected in the 36-156 inch zone for either core.

Soil pH, Organic Matter Content and Particle Size Distribution

The field in area A consisted of soil classified as a Rowdy clay loam. Our analyses (Table 6) showed the soil samples to be moderately acidic (pH 4.8 to 5.8), with a high organic matter content (6.10 to 12.3%), and similar percentages of sand (26.4 to 33.1%), silt (29 to 37%) and clay (23.2 to 37.2%).

Table 5. Concentrations of fenamiphos residues in soil core samples collected on December 2, 1986 from an unharvested portion of a lily bulb field in Area D.

Approximate depth (inches)	Fenamiphos (ppm) expressed as total residue and fenamiphos (F), sulfoxide (SO), and sulfone (SO2)							
	Core 1				Core 2			
	Total	F	SO	SO2	Total	F	SO	SO2
0 - 6	0.47	0.23	0.16	0.08	0.08	0.02	0.02	0.04
6 - 12	1.75	1.11	0.33	0.31	0.67	0.31	0.24	0.12
12 - 18	0.68	0.28	0.22	0.18	0.70	0.31	0.27	0.12
18 - 24	ND[a]	ND	ND	ND	1.13	0.51	0.54	0.08
24 - 30	0.01	0.01	ND	ND	0.25	0.11	0.14	ND
30 - 36	0.02	0.01	0.01	ND	ND	ND	ND	ND
36 - 156[b]	ND	ND	ND	ND	ND	ND	ND	ND
% of Total		56	25	19		45	42	13

a. None detected; minimum detectable level was 0.01 ppm.

b. Samples were collected at 6 inch intervals, all were none detected.

Table 6. Measurement of pH, organic carbon content, and particle size characteristics of soil samples collected from one lily bulb field in Area A on August 19, 1986.

Approximate	CORE 1					CORE 2				
depth (inches)	pH	% Organic	% Sand	% Silt	% Clay	pH	% Organic	% Sand	% Silt	% Clay
0 - 6	5.8	11.3	28.5	35	25.2	5.7	10.7	33.1	32	24.2
6 - 12	5.1	12.3	26.5	33	28.2	4.9	10.9	28.9	34	26.2
12 - 18	4.9	11.9	30.9	29	28.2	5.1	8.9	28.9	34	28.2
18 - 24	5.0	10.0	29.8	37	23.2	5.1	8.6	29.2	34	28.2
24 - 30	5.1	9.4	26.4	34	30.2	4.8	8.6	29.2	34	28.2
30 - 36	5.2	8.1	29.7	33	29.2	5.1	8.5	28.3	33	30.2
36 - 42	5.2	6.0	29.8	30	34.2	5.2	7.4	26.4	36	30.2
42 - 48	--[a]	--	--	--	--	5.3	NA[b]	26.8	36	37.2

a. Not sampled.
b. Not analyzed.

Soil samples from the field in area B (Table 7), classified as Russ silt loam, had a high silt content (35-48%), with lesser amounts of clay (25.2 to 44.2%) and sand (9.8 to 39.8%). The soil samples were moderately acidic (pH 4.8 to 5.9) with a lower organic matter content (1.1 to 4.7%).

The field in area D consisted of soil classified as a Ferndale fine sandy loam. The pH range was near neutral (6.3 to 7.5) for all but a few soil samples (Table 8). Organic matter content was low throughout the five cores with only a few values ranging above 2%. Most of the soil samples had a high percentage of sand and lesser percentages of silt and clay although the particle size distribution differed with depth in each of the cores.

Table 7. Measurement of pH, organic matter content, and particle size characteristics of soil samples collected from one lily bulb field in Area B on August 19, 1986.

Approximate	CORE 1					CORE 2				
depth (inches)	pH	% Organic	% Sand	% Silt	% Clay	pH	% Organic	% Sand	% Silt	% Clay
0 - 6	5.9	4.9	12.9	46	36.2	5.1	5.2	20.6	41	33.2
6 - 12	4.8	5.7	12.1	48	34.2	5.2	4.9	23.9	37	34.2
12 - 18	5.1	4.3	15.5	44	36.2	5.1	3.7	20.1	40	36.2
18 - 24	5.0	3.4	18.4	41	37.2	5.1	2.8	24.8	40	35.2
24 - 30	5.2	2.6	22.8	41	36.2	5.3	1.9	27.8	42	30.2
30 - 36	5.2	1.4	9.8	46	44.2	5.3	1.4	9.8	46	44.2
36 - 42	5.3	1.1	19.8	46	34.2	5.2	1.1	39.8	35	25.2

Table 8. Measurement of pH, organic matter content, and particle size characteristics of soil samples collected from five different areas of a lily bulb field in Area D on August 19, 1986.

Approximate depth (inches)	CORE 1					CORE 2				
	pH	% Organic	% Sand	% Silt	% Clay	pH	% Organic	% Sand	% Silt	% Clay
0 - 6	7.1	1.8	72.2	22	5.8	6.6	1.6	70.6	22	7.4
6 - 12	6.7	1.6	70.2	22	7.8	6.0	1.9	69.6	22	8.7
12 - 18	6.0	1.7	69.2	24	6.8	6.7	1.0	75.6	18	6.4
18 - 24	6.4	1.3	73.2	21	5.8	6.6	0.8	75.6	19	5.4
24 - 30	6.9	1.5	66.2	25	8.8	6.7	0.7	61.6	33	5.4
30 - 36	6.8	0.3	94.2	5	0.8	6.6	0.6	64.6	30	5.4
36 - 42	6.9	0.1	98.2	1	0.8	6.5	0.8	65.6	29	5.4
42 - 48	6.7	0.1	NA[a]	NA	NA	6.5	1.3	58.6	34	7.4
48 - 54	6.8	0.1	NA	NA	NA	6.5	1.1	62.6	30	7.4
54 - 60	6.7	0.7	NA	NA	NA	6.6	1.8	34.6	49	16.4
60 - 66	6.9	0.8	96.0	4	0.0	6.6	1.5	39.6	49	11.4
66 - 72	6.9	0.1	96.2	2	1.8	6.7	1.3	45.6	44	10.4
72 - 78	7.1	0.3	89.2	9	1.8	6.8	1.1	50.6	41	8.4
78 - 84	7.2	0.3	NA	NA	NA	6.8	1.4	28.6	53	18.4
84 - 90	6.9	0.1	96.2	3	0.8	6.9	1.0	40.6	47	12.4
90 - 96	7.4	0.6	86.2	10	3.8	7.0	1.3	26.6	56	17.4
96 - 102	7.2	0.2	86.2	11	2.8	6.9	1.1	37.6	49	13.4
102 - 108	7.1	0.3	NA	NA	NA	7.0	1.2	45.6	43	11.4

a. Not analyzed.

Table 8. (Continued)

Approximate depth (inches)	CORE 3					CORE 4				
	pH	% Organic	% Sand	% Silt	% Clay	pH	% Organic	% Sand	% Silt	% Clay
0 - 6	7.1	3.1	58.5	31	7.4	6.8	1.8	46.6	42	11.4
6 - 12	6.9	1.7	77.6	17	5.4	6.3	2.2	44.6	43	12.4
12 - 18	7.4	0.6	82.6	12	5.4	5.7	2.3	43.6	44	13.4
18 - 24	7.5	0.5	90.6	8	1.4	6.3	2.1	37.6	47	15.4
24 - 30	7.2	0.9	80.6	14	5.4	6.6	1.5	43.6	43	13.4
30 - 36	--[a]	--	--	--	--	6.9	1.1	56.6	34	9.4
36 - 42						7.0	1.3	54.6	37	8.4
42 - 48						6.8	1.7	71.6	21	7.4
48 - 54						7.0	0.4	90.6	8	1.4
54 - 60						7.1	0.3	93.6	5	1.4
60 - 66						6.9	0.2	98.6	1	0.4
66 - 72						6.8	0.2	98.6	0	1.4
72 - 78						7.1	0.2	97.6	1	1.4
78 - 84						7.0	0.2	98.6	0	1.4
84 - 90						6.8	0.2	99.6	0	0.4
90 - 96						6.9	0.2	99.6	0	0.4
96 - 102						6.8	0.1	99.6	0	0.4
102 - 108						6.8	0.1	98.6	1	0.4

a. Not sampled.

Table 8. (Continued)

Approximate depth (inches)	CORE 5				
	pH	% Organic	% Sand	% Silt	% Clay
0 - 6	6.9	3.5	46.1	38	12.4
6 - 12	6.2	1.6	51.6	36	12.4
12 - 18	6.0	1.4	52.6	34	13.4
18 - 24	6.2	0.7	64.6	26	9.4
24 - 30	6.4	0.5	72.6	22	5.4
30 - 36	6.6	0.4	82.6	14	3.4
36 - 42	6.6	0.6	58.6	31	10.4
42 - 48	6.8	0.7	78.6	16	5.4
48 - 54	6.9	0.4	76.6	17	6.4
54 - 60	7.0	0.7	42.6	43	14.4
60 - 66	7.1	0.8	26.6	56	17.4
66 - 72	7.1	0.4	28.6	57	14.4
72 - 78	7.1	0.6	60.6	30	9.4
78 - 84	7.1	0.1	88.6	8	3.4
84 - 90	7.0	0.5	75.6	19	5.4
90 - 96	7.1	0.4	56.6	35	8.4
96 - 102	7.2	0.4	48.6	40	11.4
102 - 108	7.2	0.4	24.6	60	15.4

DISCUSSION

This monitoring study was conducted to determine the persistence and downward movement of fenamiphos in soils of the lily bulb production area located in the Smith River Plains area of Del Norte County. Fenamiphos use has become widespread since the first applications were made in the fall of 1983 following the cancellation of aldicarb use on bulbs. This pesticide was considered to have a low mobility in soil and, therefore, not likely to leach to any great extent in soils (3). However, the practice of applying fenamiphos in a concentrated band 4 to 8 inches deep in the planting furrow created an ideal situation for downward leaching of the pesticide following rainfall or irrigation. Further, the coarse soil conditions, high winter rainfall and periodic shallow ground water tables described by the NCRWQCB for the study area added to the high risk conditions for potential contamination by fenamiphos.

Our results show that fenamiphos residues persisted for 9 to 14 months in the three lily bulb fields that were monitored, and during that time, residues moved well below the zone of application. Rainfall data obtained from the Crescent City airport, approximately 7 miles southwest of the study area, showed that heavy rainfall during the 1985-86 season probably contributed to the leaching of fenamiphos. More than 42 inches of rain fell between the time fenamiphos was applied in October and the first soil cores were collected in March. An additional 31 inches and 5 inches fell before the next sets of cores were sampled in July and August, respectively. Thus, a total of about 78 inches of rain and an unspecified amount of irrigation water was applied to the study area over a 10 month period.

Of the four cores collected from areas A and B in August, fenamiphos residues were found in the deepest (42-48 inches) segments of three of the cores, suggesting that residues might have been present deeper in the soil. Soils in these fields were silt or clay loams, moderately acidic, and had a relatively high organic matter content, especially in area A. All of these factors could account for the longevity of fenamiphos since it is considered to be stable at neutral to slightly acidic conditions (3) and organic matter tends to bind organic pesticide molecules in the soil.

After more extensive and deeper coring in August in area D located near the Smith River, it became apparent that fenamiphos was leaching in that field. The maximum depth at which fenamiphos was detected varied among the five areas of the field that were sampled, but it was found 108 inches (9 feet) deep at two locations and 90 inches (7-1/2 feet) deep at a third. The sandy loam soil in this field was in the neutral pH range and contained little organic matter. Both of these factors would favor the persistence of fenamiphos in soil and make it available for leaching. The particle size (texture) analyses of the soil cores showed that there were large differences in the soil profiles between the five areas sampled. However, fenamiphos was found throughout soil cores having vastly different sand, silt, or clay contents at various depths. Fenamiphos residues were still present in soil cores collected from the field in December, approximately 14 months after treatment, however, the chemical was found at maximum depths of only 30 to 36 inches.

Once fenamiphos has been added to soil, oxidation to fenamiphos sulfoxide is reported to occur rapidly followed by a much slower degradation to fenamiphos sulfone (3,4,5). Fenamiphos sulfoxide has also been reported to be the most persistent and mobile in soil (5,6). The results of our study were contradictory to those findings. For example, in all soil cores collected in August, 9 months post application, the total fenamiphos residues were comprised of a high (44 to 81%) proportion of the parent compound and lesser amounts (9 to 49%) of the sulfoxide. Further, the parent compound was present in most of the positive samples collected, was the only compound present in the deepest positive samples from four of the nine cores, and was also present along with the sulfoxide and sulfone in the deepest positive samples from four additional cores. These findings suggest that fenamiphos residues were influenced differently by the soil and climatic conditions present in Del Norte County. However, the apparent deep movement of the parent compound into the soil also suggests that contamination may have occurred during the soil coring process. This possibility will be investigated in subsequent monitoring studies.

The results of soil sampling over several months demonstrated that fenamiphos and its two oxidation breakdown products persisted for long periods in the soils of Del Norte County. This persistence and apparent mobility of the compounds through the soil, together with the high rainfall that occurs soon after application, may pose a threat to groundwater supplies in certain areas.

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APPENDIX I

ANALYTICAL METHOD FOR NEMACUR IN SOIL

DEPARTMENT OF FOOD AND AGRICULTURE

CALIFORNIA DEPT. OF FOOD & AGRIC.
ENVIRONMENTAL MONITORING SECTION
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Original Date:November 7, 1985
Supercedes:NEW
Current Date:November 7, 1985
Method #:

Nemacur Residues in Soil

SCOPE:
This method has been developed and used for the analysis of Nemacur, Nemacur Sulfoxide, and Nemacur Sulfone in soil.

PRINCIPLE:
Nemacur and its metabolites are extracted from the soil with a hexane-acetone (1:1) solution. The solution is evaporated to dryness and redissolved in ethyl acetate. A portion of the extract is prepared for the GLC analysis of Nemacur. The remaining extract was evaporated to dryness and redissolved in acetonitrile:water (20:80). This portion was then analyzed by HPLC for the metabolites.

REAGENTS AND EQUIPMENT:

1. Acetonitrile, HPLC grade
2. Ethyl Acetate, Pesticide grade
3. Water, HPLC quality, filterd
4. Balance - Mettler PL 1200 - Mettler Instrument Corp. Hightstown,N.J.
5. Micro-Mate Syringes 10cc - Popper and Sons Inc. New Hyde Park, N.Y.
6. 500ml flat-bottom boiling flasks
7. Funnels, 60 degree short stem, 3-4 inch diameter.
8. Graduated conical centrifuge tube - 15ml
9. Bottles, 500ml amber wide-mouth with teflon lined lid - Qorpak.
10. Whatman #4 filter paper or Sharkskin - 12.5cm
11. Rannin HPLC Prefilters - 0.2 micron
12. Assorted glassware for measuring and dispensing reagents as required.
13. Reverse phase HPLC with UV detector.
14. Meyers N-EVAP - Organomation Associates Incorporated Northborough, Ma.
15. G-10 Gyrotory (R) Shaker - New Brunswick Scientific Co.,Inc. (with CE-250S clamps) Edison, N.J.
16. Thermolyne Vortex Maxi Mixer II - Sybron Corporation Dubuque, Iowa.
17. 57mm aluminum weighing dish - Fisher Scientific San Francisco, Ca.

ANALYSIS:

Exraction:

1. Soil core samples were thawed at room temperature or in the refridgerator overnight and mixed well.

2. Weigh 15-20 gram of sample in an aluminum dish and place in an oven at 110°C for at least six hours for determining soil moisture.

3. Weigh 50 grams of soil into a 500ml wide-mouth amber bottle. Add ~ 100 gram anhydrous sodium sulfate to sample and mix well. Add 60mls of hexane-acetone (1:1), cover with foil, cap and shake vigorously for ten seconds.

4. Place on Gyrotory shaker for twenty minutes at 230 rpm.

5. Let sample set for fifteen minutes after removal from the shaker.

6. Decant solvent from sample through funnel, lined with filter paper and and filled with ~ 100 gram anhydrous sodium sulfate, into a 500 ml boiling flask. Rinse sodium sulfate with 20 ml hexane-acetone.

7. Add another 60 ml of hexane-acetone (1:1) to each sample, recap and repeat steps 4, 5, and 6 two more times.

8. On the last extraction decant the organic layer and finally the soil into the funnel. Rinse the sample bottle with 20 ml of hexane-acetone and pour through funnel.

9. Rotoevaporate at 40°C under 15 inches vacuum until almost dry.

10. Using ethyl acetate, quantitatively transfer residues to a 15 ml graduated centrifuge tube and bring to final volume of 5 ml under nitrogen (50°C) on the N-EVAP.

GLC Preparation:

1. Place samples on the Maxi Mixer for 20 seconds.

2. Remove 2 ml of sample and place in auto sampler vial for GLC analysis.

HPLC Preparation:

1. Return the remaining 3 ml of sample to the N-EVAP (@50°C) and evaporate the ethyl acetate to dryness.

2. Add 2 ml (with volumetric pipette) of 20% acetontrile-water to redissolve the residues.

3. Place samples on Maxi Mixer for 20 seconds then sonicate for 2 minutes.

4. Place on Maxi Mixer for 30 seconds then transfer to a 5 cc syringe. Filter through a 0.2 micron HPLC prefilter into an autosampler vial ready for HPLC analysis.

HPLC CONDITIONS:

Perkin Elmer Series 4 HPLC with ISS automatic sampler and column oven, or equivalent. An ultraviolet detector, Kratos SF 769Z at a wavelength of 220 nanometers.

Column:
Sepralyte cyclohexal (CH), 5 micron, 4.6mm i.d. x 25cm
(Analytichem International)

Flow conditions:
Equilibrium - 1.5ml/minute for seven minutes of
15% acetonitrile / 85% water

Gradient - Flow 1.5ml/minute
2 minutes @ 15% acetonitrile / 85% water
6 minute @ 25% acetonitrile / 75% water
11 minutes @ 40% acetonitrile / 60% water
8 minutes @ 60% acetonitrile / 40% water
3 minutes @ 75% acetonitrile / 25% water

Oven Temperature = Ambient

Injection Volume = 100 microliters

GLC CONDITIONS:

Varian 3700 equiped with a Thermionic Specific Detector and a Hewlett-Packard 7672A auto sampler.

Injector: Splitless; 210°C

Detector: 260°C
Bead; 470
Hydrogen; 25 psi

Temperature Program: Initial temperature; 130°C for 1 minute.
Program Rate; 20°C per minute.
Final temperature; 230°C for 3 minutes.

Column: Hewlett-Packard HP-1(crosslinked)
100% Dimethyl polysiloxane (Gum)
10m x 0.53 x 2.65 micron
Carrier: Helium 12 ml/ minute

CALCULATIONS:
Report data in ppm.

$$\%\ \text{Moisture} = 100 - \frac{(\text{dry weight soil})\ (100\%)}{(\text{wet weight soil})}$$

$$\text{PPM} = \frac{(\text{peak ht sample})(\text{ng std injected})(\text{sample final volume ml})(100)}{(\text{peak ht standard})(\text{ul injected})(\text{g of sample})(100 - \%\text{moisture})}$$

DISCUSSION:

REFERENCES:

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APPENDIX II

METHOD FOR SOIL ORGANIC MATTER DETERMINATION

ORGANIC MATTER (O.M.)

Dichromate reduction

EQUIPMENT

Soil grinder of non-ferrous material (mullite mortar and pestle)
0.5 mm screen (40-60 mesh)
Erlenmeyer Flasks, 500 ml
Thermometer, 200°C
Bunson burner or electric hot plate

Reagents

1. Potassium dichromate solution, 1.0 N. Dissolve 49.04 g of dry reagent grade potassium dichromate, ($K_2Cr_2O_7$) in distilled water and dilute to 1 liter.

2. Sulfuric acid-silver sulfate solution. Dissolve 25 g of reagent grade silver sulfate (Ag_2SO_4) in 1 liter of reagent grade concentrated 36 N sulfuric acid.

3. Ortho-phenanthroline ferrous sulfate indicator solution. Dissolve 1.485 g of 1,10-phenanthroline monohydrate (Eastman Kodak No. 3239) and 0.695 g of ferrous sulfate ($FeSO_4$) in distilled water and dilute to 100 ml.

4. Ferrous sulfate solution 0.5 N. Dissolve 140 g of ferrous sulfate ($FeSO_4 \cdot 7H_2O$) in distilled water, add 15 ml of reagent grade concentrated H_2SO_4. Cool and dilute to 1 liter.

Procedure

1. Grind soil to pass 0.5 mm screen.

2. Weigh 5.00 g of soil into 500 ml flask. We used from 1 to 5 grams of soil.

3. Add 10 ml Reagent 1 and then 20 ml Reagent 2; both reagents are conveniently dispensed from burettes.

4. Mix well by swirling; insert thermometer and heat gently over burner or on hot plate to reach a temperature of 150°C in one minute. Swirl contents continuously while heating to avoid local super-heating and consequent decomposition of dichromate. (The heating time and temperature must be adhered to.)

5. Remove from heat and cool.

6. Add approximately 200 ml of water.

7. Add 3-4 drops of Reagent 3.

8. Titrate with Reagent 4 to sharp red endpoint. Record ml titration as "A".

9. Standardize Reagent 4 for each set of samples by running 10 ml of Reagent 1 through the procedure. Record titration as "B".

10. Calculate percent organic matter.

Calculations

$$\text{Percent Organic Matter} = (B-A) \times \frac{10}{B} \times 0.58/\text{g of soil used}$$

Remarks

If more than 80% of the dichromate is reduced, "A" < 4 ml, the determination should be repeated using less soil.

The factor, 0.58 is derived from:

the milliequivalent weight of carbon, 0.003;

the assumption that this method gives 89% recovery of organic carbon in soils;

the assumption that the organic matter of soils contains 58% carbon.

If difficulty is experienced in obtaining a distinct endpoint, it will be helpful to filter the digest at Step 5: After cooling, add 100 ml of water, filter through Whatman No. 2 on a Buchner funnel, washing with another 100 ml of water. Then proceed with Step 7.

APPENDIX III

METHOD FOR SOIL PARTICLE SIZE ANALYSIS

PROCEDURE

The procedure in detail is as follows: Dissolve 50 g. Calgon in a liter of distilled water. Pour 100 cc. of this solution into a pint jar. Add 50 g. of air-dry soil (100 g. in the case of very sandy soil). Mix thoroughly and let stand in covered jar overnight or 15 to 20 hours. Then wash contents into the soil cup (Figure 1) with distilled water. Fill the cup with water to within 3 inches from the top. Connect cup to the dispersing machine and stir for 2 minutes. Disconnect cup and wash contents into soil cylinder using a water jet from the plastic bottle. Fill soil cylinder to the liter mark. Bring cylinder and contents to 68°F. by placing in a water bath. Remove cylinder and close mouth with rubber stopper. With right hand holding and pressing on the stopper, and left hand holding the bottom of the cylinder, turn cylinder completely upside down and back 20 times. Return cylinder to water bath and immediately start a timer or stop watch. Quickly put 3 drops of amyl alcohol on top of soil suspension column to dissipate froth and at 15 seconds gently place hydrometer in the soil suspension column and prepare to take a hydrometer reading at 40 seconds. Remove the hydrometer and wash it. The last hydrometer reading is to be taken after sedimentation has continued for exactly 2 hours.

Temperature affects the hydrometer readings and since the hydrometer has been calibrated at 68°F. the soil cylinder with contents should be kept in a bath at this temperature or an attempt should be made to work close to this temperature. In fact, the ideal place to conduct mechanical analyses of soils by the hydrometer method is in a 68°F. constant temperature room. Where temperature correction has to be made, multiply differences in temperature above or below 68°F. by a factor or 0.2. The product above 68° is added to the hydrometer reading and the product below 68° is subtracted. Use of the correction factor is permissible only within the temperature range 60 to 76°F.

When floating in a 0.5% solution of Calgon (100 cc. 5% solution diluted to 1 liter) the hydrometer has a stem reading of 6.5. This reading must be subtracted from every hydrometer reading obtained with soil suspensions prepared in the described manner.

To calculate the amounts of combined sands, of silt, and of clay as determined by the hydrometer method the procedure is as follows for the U.S. Department of Agriculture soil particle size classification:

The corrected hydrometer reading at the end 40 seconds is divided by the amount of dry soil taken and multiplied by 100. This result is the percentage of material still in suspension at the end of 40 seconds. This percentage is subtracted from 100 and the result is this percentage of material that settled out at the end of 40 seconds, which represents all the sand in the soil (2.00 - 0.05 mm). The corrected hydrometer reading at the end of 2 hours is also divided by the dry weight of the soil and multiplied by 100. The result is percentage of material still in suspension at the end of 2 hours and is the clay (below 0.002 mm). The percentage of silt (0.05-0.002 mm) is obtained by difference.

At the conclusion of the 2-hour hydrometer reading, the suspension is washed on a No. 300 sieve. That portion retained by the sieve is dried and analyzed on a set of sieves consisting of one each of No. 20, 40, 60, 140, and 200.